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Lessons Learned: Quality Assurance and Explosives Analyses

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DOD Environmental Monitoring & Data Quality Workshop

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Agenda

- Scoping considerations – the evolution of a DID
- Sampling and Analysis Plan review
- Lessons learned regarding confirmation analyses

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Munitions Constituents "DID"

DATA ITEM DESCRIPTION

Title: Munitions Constituents Chemical Data Quality Deliverables

Number: MR-005-10 **Approval Date:** 20031201
AMSC Number: **Limitation:**
DTIC Applicable: No **GIDEP Applicable:** No
Office of Primary Responsibility: CEHNC-ED-CS-P
Applicable Forms:
Use/Relationship: Munitions Constituents (MC) Chemical Data Quality Deliverables will be used to describe planning and results of sampling and analysis, quality assurance/quality control, laboratory qualification, data acquisition/data reporting, and chain-of-custody when environmental samples are required for Munitions Response or other munitions related projects.

- Replaces majority of SOW language on sampling and analysis
- Based on Unified Federal Guide Specifications
- Represents 3 years of lessons learned in explosives analysis contracting
- Available at http://www.hnd.usace.army.mil/oew/didsindex.asp

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DID Highlights

1.3.1 Laboratory Validation Requirements

The Contractor shall propose the minimum number of laboratories that can attain or have attained U.S. Army Corps of Engineers (USACE) validation in accordance with EM 200-1-1 and consistent with contract required chemical data quality. The Contractor may propose laboratories that shall subsequently be validated by the USACE, or select currently validated USACE laboratories. The laboratory must hold applicable state accreditations. The Contractor shall identify all proposed project laboratories in the proposal and in the sampling and analysis plan (SAP). If a proposed analytical laboratory cannot meet specified analytical requirements or achieve the required validation, the Contractor shall select another laboratory. If not currently validated, the USACE laboratory validation process requires a nominal 120-day process. Samples may not be subcontracted to another laboratory without approval of the CO. If a subcontractor laboratory must be used, the subcontractor laboratory must meet all requirements for validation and accreditation, as well as project-specific SAP requirements.

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DID Highlights (Cont'd)

2.7.1 Laboratory Analytical Requirements

The Contractor shall provide the specified chemical analyses by the Contractor's laboratory. The Contractor shall provide chemical analyses to achieve the project DQO for all parameters specified by the methods. To give the USACE programs the greatest flexibility in the execution of its projects, the EPA 530/F-93/004 methods are generally the methods employed for the analytical testing of environmental samples. These methods are flexible and shall be adapted to individual project-specific requirements. Method performance must be in accordance with EM 200-1-3, Appendix I requirements, unless variances are specifically approved in the SAP. The requirement for the laboratory to provide quantitative second column confirmation for explosives per EM 200-1-3/SW8000B (i.e., five-point calibrations must be performed for each target analyte for the primary and confirmatory columns and quantitative results for each column must be reported) will not be waived. Based upon project requirements, exceptions will be considered for the following coeluting pairs: 2-A-DNT/4-A-DNT, 2-NT/4-NT, and 2,4-DNT/2,6-DNT.

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Sampling and Analysis Plan Review – FSP

- Confirm that samples are composites (most explosives-related sampling should be taken as composite samples)
- Confirm that appropriate safety precautions are in place if sampling in an ordnance contaminated area; if you aren't qualified to make this assessment, ask someone who is!

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Sampling and Analysis Plan Review – QAPP

- Look at everything QAPP says about SW8330!
- Contaminants of Concern
  - Do you need PETN or NG?
- Sample Preparation
  - Representative sampling shouldn't end when the sample enters the laboratory.
  - SW8330 calls for mortar and pestle grinding of soil samples. Confirm that laboratory performs at least this level of sample homogenization.
  - If samples may contain % HE, field screen and/or lab screen
  - For water, SPE is recommended. Verify laboratory's method of choice.
  - Drying should be performed at room temperature.

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Sampling and Analysis Plan Review – QAPP (Cont'd)

- Laboratory Analysis
  - Look at relationship between MDLs and PQLs (many labs use PQLs that are very close to PQLs for explosives)
  - Confirm LCS and MS/MSD recovery requirements are reasonable (and compliant with EM 200-1-3 and/or DoD QSM, as required by SOW)
  - Confirm that laboratory is performing quantitative confirmations.
  - If at all possible, review SOP. Look for descriptions of coelution problems. Retention time tables are an easy way to find out.
  - If it isn't clear from the QAPP, ask if they have coelution issues.
  - Ask how the laboratory quantitates affected compounds if there are coelutions.
  - When in doubt, ask to review calibration chromatograms.

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Sampling and Analysis Plan Review – QAPP (Cont'd)

- **Data Verification/Validation**
  - **Determine basis for review. (IAW Functional Guidelines doesn't really work for HPLC methods)**
  - **If there are coelution issues, data verification/validation sections should describe how the data reviewer will address those issues.**
  - **Recommend that chromatograms for any positive results for explosives be reviewed by contractor and spot-checked by government chemist**

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Lessons Learned – Confirmations

- If you have positive explosives results, particularly in situations where they seem unlikely, strongly recommend looking closely at the confirmation data.
- This extends to looking at the chromatograms.
- Following case study is from a final document received for review by the OE CX.
- Data review by laboratory and contractor was documented.

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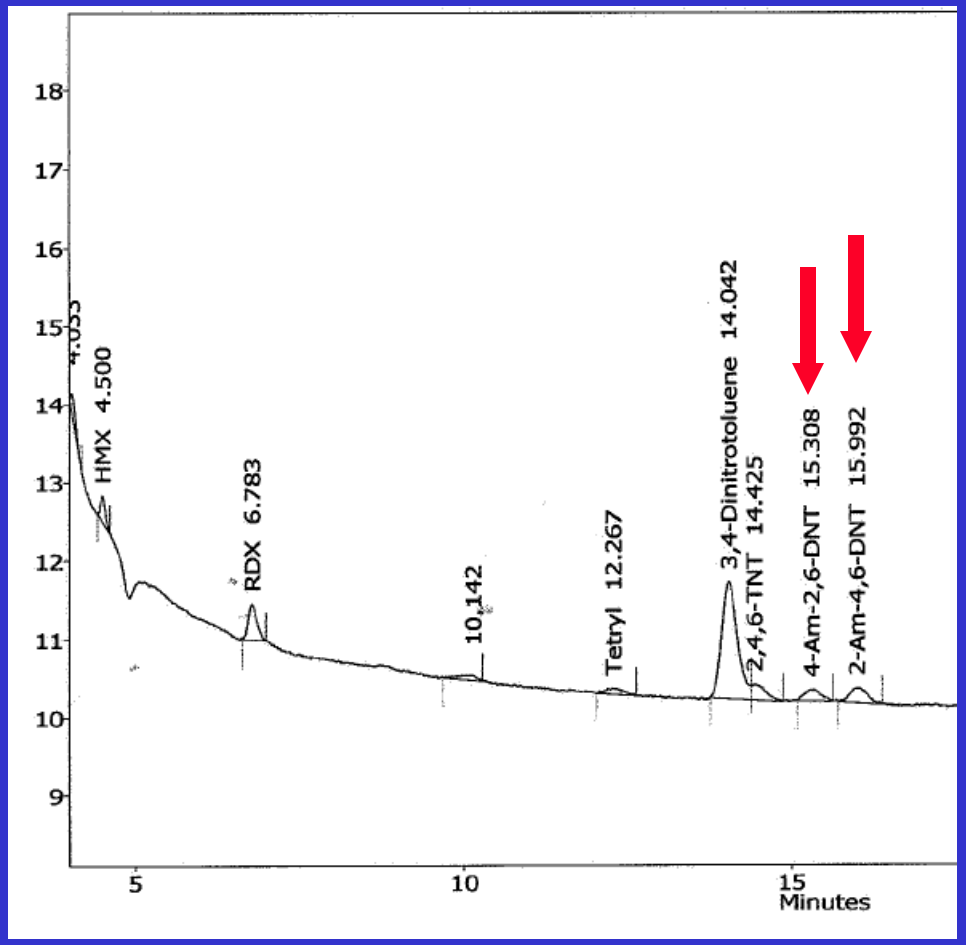
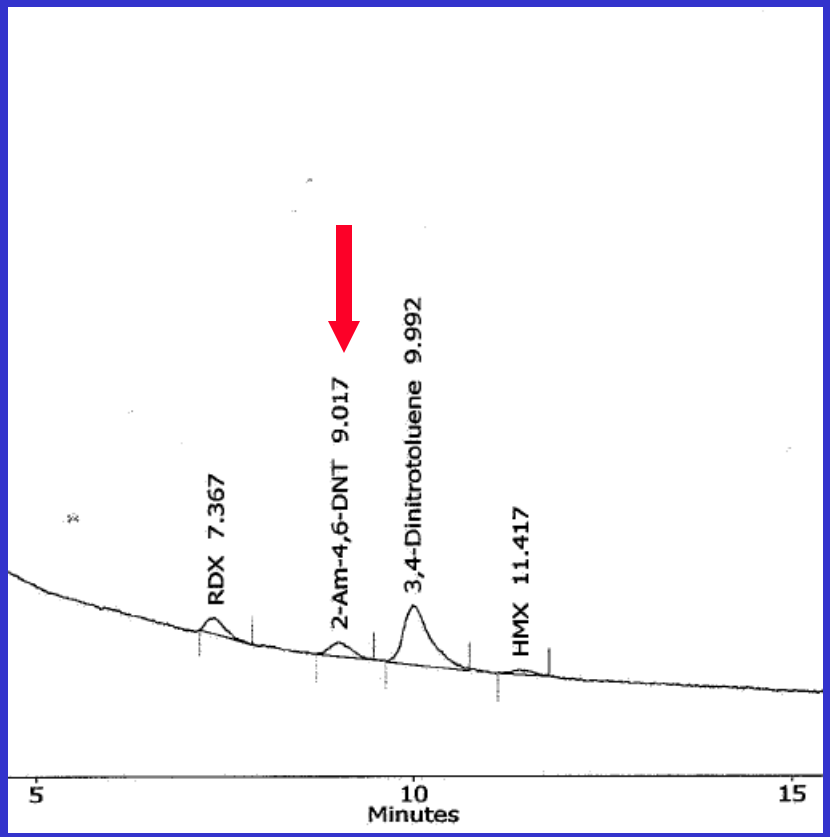
Am-DNT Comparison – Chromatogram



Primary Column (C-18)



Secondary Column (CN)

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Am-DNT Comparison – Data

Primary Column

#	Peak Name	Ret.Time(min)	Area	Ave. RF	ESTD Conc.(ppb)
2	HMX	4.500	1819	68.7	26.5
3	RDX	6.783	4165	79.9	52.1
--	1,3,5-TNB	9.067	0	0.0	0.0
--	1,3-DNB	11.117	0	0.0	0.0
5	Tetryl	12.267	1553	166.1	9.3
--	Nitrobenzene	12.808	0	0.0	0.0
6	3,4-Dinitrotoluene	14.042	25079	119.4	210.0
7	2,4,6-TNT	14.425	2908	210.6	13.8
8	4-Am-2,6-DNT	15.308	2552	147.0	17.4
9	2-Am-4,6-DNT	15.992	3517	213.6	16.5
--	2,6-DNT	16.733	0	0.0	0.0
--	2,4-DNT	17.400	0	0.0	0.0
--	2-Nitrotoluene	20.658	0	0.0	0.0
--	4-Nitrotoluene	22.325	0	0.0	0.0
--	3-Nitrotoluene	23.975	0	0.0	0.0

Secondary Column

#	Peak Name	Ret.Time(min)	Area	Ave. RF	ESTD Conc.(ppb)
--	Nitrobenzene	5.233	0	0.0	0.0
--	1,3-DNB	6.042	0	0.0	0.0
--	1,3,5-TNB	6.808	0	0.0	0.0
--	2-NT/3-NT/4-NT	7.100	0	0.0	0.0
1	RDX	7.367	3926	73.9	53.1
--	2,4-DNT	8.092	0	0.0	0.0
--	2,6-DNT	8.200	0	0.0	0.0
2	2-Am-4,6-DNT	9.017	4123	153.7	26.8
--	4-Am-2,6-DNT	9.125	0	0.0	0.0
3	3,4-Dinitrotoluene	9.992	21004	95.5	219.9
--	2,4,6-TNT	10.392	0	0.0	0.0
4	HMX	11.417	1314	52.4	25.1
--	Tetryl	16.467	0	0.0	0.0

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TNT Comparison – Data

Primary Column

#	Peak Name	Ret.Time(min)	Area	Ave. RF	ESTD Conc.(ppb)
2	HMX	4.500	1819	68.7	26.5
3	RDX	6.783	4165	79.9	52.1
--	1,3,5-TNB	9.067	0	0.0	0.0
--	1,3-DNB	11.117	0	0.0	0.0
5	Tetryl	12.267	1553	166.1	9.3
--	Nitrobenzene	12.808	0	0.0	0.0
6	3,4-Dinitrotoluene	14.042	25079	119.4	210.0
7	2,4,6-TNT	14.425	2908	210.6	13.8
8	4-Am-2,6-DNT	15.308	2552	147.0	17.4
9	2-Am-4,6-DNT	15.992	3517	213.6	16.5
--	2,6-DNT	16.733	0	0.0	0.0
--	2,4-DNT	17.400	0	0.0	0.0
--	2-Nitrotoluene	20.658	0	0.0	0.0
--	4-Nitrotoluene	22.325	0	0.0	0.0
--	3-Nitrotoluene	23.975	0	0.0	0.0

Secondary Column

#	Peak Name	Ret.Time(min)	Area	Ave. RF	ESTD Conc.(ppb)
--	Nitrobenzene	5.233	0	0.0	0.0
--	1,3-DNB	6.042	0	0.0	0.0
--	1,3,5-TNB	6.808	0	0.0	0.0
--	2-NT/3-NT/4-NT	7.100	0	0.0	0.0
1	RDX	7.358	3199	73.9	43.3
--	2,4-DNT	8.092	0	0.0	0.0
--	2,6-DNT	8.200	0	0.0	0.0
2	2-Am-4,6-DNT	9.050	4163	153.7	27.1
--	4-Am-2,6-DNT	9.125	0	0.0	0.0
--	3,4-Dinitrotoluene	10.100	0	0.0	0.0
3	2,4,6-TNT	10.400	1226	149.6	8.2
4	HMX	11.442	977	52.4	18.6
--	Tetryl	16.467	0	0.0	0.0

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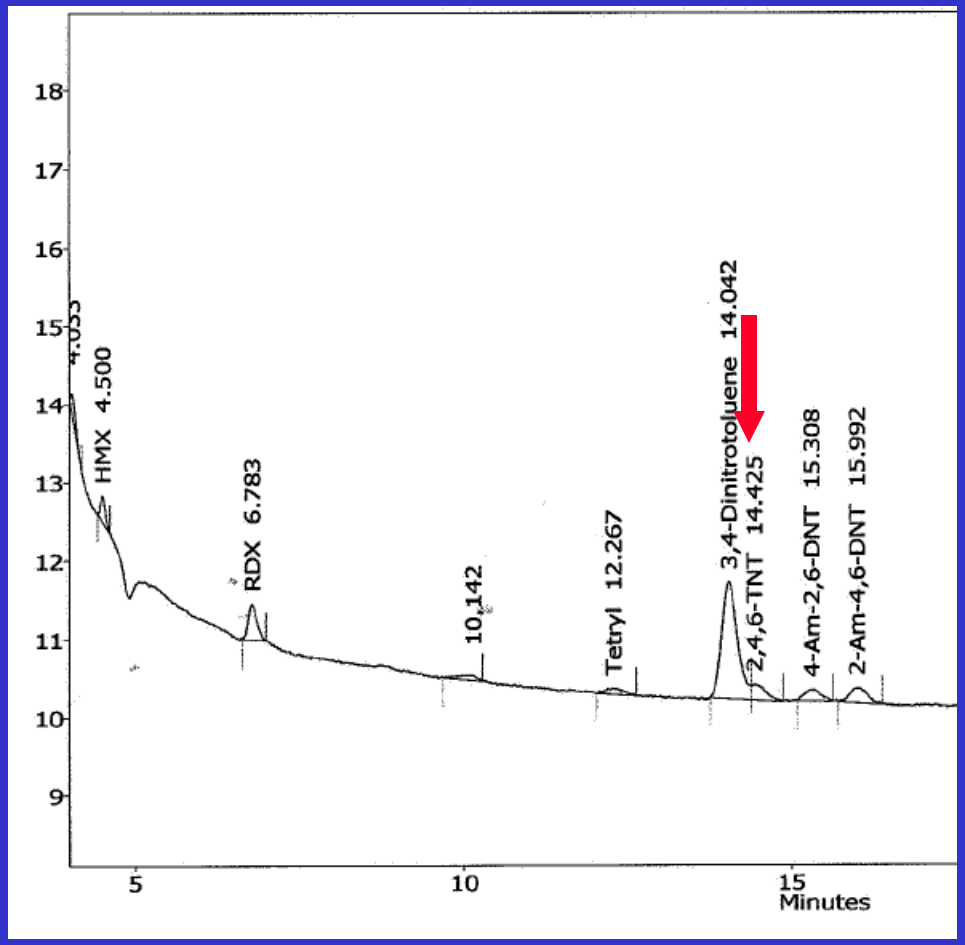
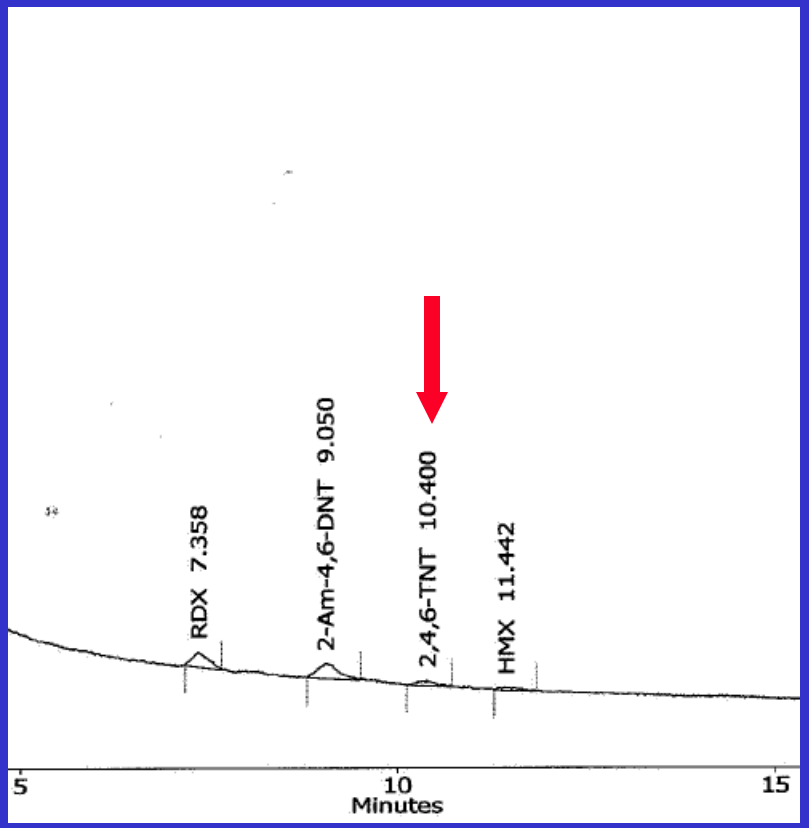
TNT Comparison – Chromatogram



Primary Column (C-18)



Secondary Column (CN)
No surrogate

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TNT and Am-DNT Comparison - Results

Primary Column Results:

- TNT – 138 µg/kg
- 4-Am-2,6-DNT – 174 µg/kg
- 2-Am-4,6-DNT – 165 µg/kg

Secondary Column Results:

- TNT – 82 µg/kg
- 4-Am-2,6-DNT – 0 µg/kg
- 2-Am-4,6-DNT – 268 µg/kg

PARAMETERS	RESULTS (ug/kg)	RL (ug/kg)	MDL (ug/kg)
HMX	260J	400	200
RDX	520	400	200
1,3,5-TNB	ND	400	100
1,3-DNB	ND	400	100
TETRYL	ND	400	100
NITROBENZENE	ND	400	100
2,4,6-TNT	140J	400	100
4-AM-2,6-DNT	170J	400	100
2-AM-4,6-DNT	ND	400	200
2,6-DNT	ND	400	200
2,4-DNT	ND	400	100
2-NITROTOLUENE	ND	400	100
3-NITROTOLUENE	ND	400	200
4-NITROTOLUENE	ND	400	100
SURROGATE PARAMETERS	**% RECOVERY**	**QC LIMIT**	
3,4-DNT	105	54-154	

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ANY QUESTIONS?

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